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JANUARY 1973

**COMPOSITE HEMISPHERE FEASIBILITY STUDY (u)
FINAL REPORT**

AD 525631

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Contract DNA001-73-C-0021

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COMPOSITE HEMISPHERE FEASIBILITY STUDY

ABSTRACT

The feasibility of applying slip casting technology to the fabrication of a complex rounded carbon matrix composite structure having a controlled metal carbide content has been investigated.

The slip casting technology is based on the use of certain controlled particle size formulations; i.e., resin, carbon/graphite powders, and reinforcing fiber to fabricate a high strength carbon matrix composite structure.

Formulations were prepared in which certain metal carbide powders, TiC, TaC and ZrC, were incorporated into the slip formulation replacing selected fractions of the carbon/graphite powders. Conditions were established for the compression molding and carbonization of Z-loaded carbon hemispheres of approximately 4" O.D. and 100 mils wall thickness. Two different Z-layered structures (50 mils each) were fabricated into a single (100 mil) hemisphere. These carbon hemispheres showed little distortion and surface cracks after heat treatment up to 1400°C.

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1. PURPOSE OF STUDY

The objective of the two month research program performed at Esso Research and Engineering Company and supported by the Defense Nuclear Agency, Washington, D.C. (Contract No. DNA-001-73-C-0021) was to demonstrate the feasibility and virtues of using certain controlled particle size formulations for the compression molding of a complex, rounded, metal carbide loaded carbon matrix composite structure.

The complex shape chosen was a hemisphere of approximately 4" O.D. The target wall thickness of hemisphere was 100 mils. In addition, the hemisphere should contain two discrete layers, each layer containing about 25 weight percent loading of different metal carbide powders and should be no more than 50 mils thick to stay within the 100 mil total wall thickness requirement.

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2. DISCUSSION AND RESULTS OF FEASIBILITY STUDY

A graphite fiber reinforced phenolic slip formulation was prepared which consisted of approximately 45 weight percent graphite powders, 25 weight percent metal carbide powders, 20-25 weight percent of a phenolic-epoxy novalac resin mixture (10 phr-epoxy-novalac) and 10 weight percent of Hercules HMS graphite fibers. The particle sizes of the graphite powders used ranged from less than 0.2 micron (μ) to greater than 20.0 μ . It was observed that a family of particle size distributions could be utilized to effect a 70 to 75 weight percent loaded formulation. Formulations were prepared which contained three different metal carbide powders. These metal carbides were TiC, TaC and ZrC and were obtained from Atlantic Equipment Engineers of Bergenfield, New Jersey. The particle sizes of these metal carbides were specified by the manufacturer to be between 1 and 5 microns.

The general procedure used to fabricate the phenolic precursors to the carbon composite hemispheres consisted of the following sequence of steps. The Hercules HMS continuous tow graphite fibers were chopped to aspect ratios (L/D) of between 500/1 and 100/1. The chopped fibers were mixed with the phenolic and epoxy resin in a methanol-ethanol solvent system. The graphite powders and appropriate metal carbide powders were added, and the viscosity of the formulation increased to the desired level by continuous mixing. Approximately 100 g. of the viscous slip formulation was charged into the hemisphere mold, "B" staged for 0.5 hours at 140°F and compression molded at 325°F and 15 tons platten pressure for one hour. After cooling, the loaded phenolic hemisphere was removed from the mold, post-cured for 2 hours at 450°F and then carbonized to 1400°C. The carbonization cycle required a total of 48 hours. The temperature of the sample was increased only about 20°C per hour up to 1000°C during the carbonization cycle. It was observed that the greatest weight loss of the hemispheres occurred between 760°C and 930°C.

Table 1 compares typical measurements, i.e., rim O.D. and I.D., also wall thickness of the typical phenolic precursor hemispheres and typical carbon composite hemispheres. These measurements indicate that both the precursor hemispheres and the carbon composite hemispheres are virtually distortion free.

Table 1

Hemisphere Measurements

	<u>Typical Phenolic Precursor Hemisphere - TaC and TiC Loaded</u>	<u>Typical Carbon Composite Hemisphere - TaC and TiC Loaded</u>
Rim O.D.	4.189 \pm 0.014 (4)	4.143 \pm 0.038 (4)
Rim I.D.	3.980 \pm 0.007 (4)	3.950 \pm 0.033 (4)
Wall Thickness	0.110 \pm 0.005 (8)	0.100 \pm 0.006 (8)

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Table 2 summarizes the properties of the carbon matrix composite hemispheres. Considering the degree of porosity in the samples, and that no precautions were taken to minimize porosity since it was indicated that void content in the samples was desirable, the property data is very reasonable. The strength properties of the Z-loaded carbon composite are comparable to commercially available bulk graphite

Table 2

Property Data

<u>Carbon Composites - Sample No.</u>	<u>Specific Gravity - (g/cc)</u>	<u>Flexural Strength - (x 10³ psi)</u>	<u>Flexural Modulus (x 10⁶ psi)</u>
1	1.786	3.7	1.1
2	1.636	2.8	1.1
3	1.666	3.7	1.1
4	<u>1.604</u>	<u>2.6</u>	<u>1.1</u>
Averages	1.673	3.2	1.1

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3. CONCLUSIONS

The objectives of the program were accomplished. Several complex, rounded, metal carbide loaded carbon composite structures were fabricated that contained two layers of different metal carbide powders. The carbon matrix hemispheres were subjected to temperatures up to 1400°C. The wall thickness of the layered hemispheres was maintained at about 100 mils. Also the carbon matrix composite hemispheres, by rim O.D. and I.D. measurements, showed little or no distortion and dimensional instability.

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	Graphite Powders						
	Graphite Fiber						
	Carbonization						
	Slip Casting						

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